Selected Parts of a Hypothetical Paper for the

International Journal of Thermophysics

Oscillating Cup Viscosity Measurements of Aluminum Alloys: A356 and 319 [1]

Deming Wang [2] and R. A. Overfelt [2, 3]

Abstract

An oscillating cup viscometer was developed at Auburn University to measure the viscosities of molten metals. Previous experiments established the capability of the apparatus to characterize the viscosities of molten nickel-based superalloys. However, modifications to the instrument and its theoretical analysis were required for reliable measurements on molten aluminum alloys, presumably due to their lower densities and lower viscosities. The theoretic literature for the fluid flow inside an oscillating cup is reviewed and a working equation without any correction factor is developed for the improved viscometer. Some design parameters of the viscometer which directly affect the accuracy of viscosity estimation by using the working equation are discussed. A special vertical furnace was adopted to uniformly heat a longer cylinder sample (10 mm inner diameter and 120 mm long) with temperature difference over the sample length of less than 2 ºC. The measuring procedure was also improved to get more accurate motion parameters. It is estimated that the working equation and improved instrument exhibit an error less than 4%. In addition, applications and experimental data are presented for pure aluminum and two aluminum alloys A356 and A319.

KEY WORDS: aluminum alloys; molten metals; oscillating cup; viscosity.

1 Introduction

The oscillating cup viscometer has become a dominant technique to measure the viscosities of high temperature liquids[1-3]. In the viscometer, a high temperature liquid (such as a molten metal) is contained within a crucible suspended by a wire to form a torsional pendulum, which induces torsional oscillation motion. This motion is damped primarily by viscous dissipation within the viscous liquid inside the crucible. The viscosity of the liquid can be calculated by an analytical or numerical solution of the equations of motion of the oscillating cup system. The principal advantages of this technique over others are its mechanical simplicity and the ability to measure the time period and amplitude decay with great precision.

Since the1960s, a number of successful viscometers and their working equations have been developed to measure the viscosities of liquids at high temperature [4-6]. But, there are still large discrepancies between laboratories, sometimes amounting to 50%. It is commonly considered that the errors come from the different kinds viscometers and viscosity estimation methods. Further study of the principle of viscometers and their working equations is still very important to improve these measurement techniques and obtain reliable viscosity data for science and industry.

Roscoe[7-8] proposed an approximate method to calculate the viscosity from the measured motion parameters. In the method, a correction factor from a known viscosity material is needed to calibrate the measured results. Advantages of the method are that it is simple to use and easy to understand; so this method is still often employed [2]. Kestin and Newell [9] and Beckwitt and Newell [10] provided another analytic method and working equation to calculate viscosities of liquids from measured the oscillation parameters. This method does not need any correction factor since an exact solution of the equation motion of oscillating cup systems is given. One of the primary advantages is the method eliminates

calculation error from mathematical process. For some oscillating cup viscometers, calculation error is less than 0.01%. So, it is preferred to be accepted by many investigators. Ohta and et. [11] used the method for their viscometer with oscillation spheric body. Torklep and Oye [12] also used it for their new generation oscillation cup viscometer and they presented a set of simplified calculation formulae. Nunez and al.[3] also used the method for their new high-temperature viscometer to measure the viscosity of molten salts. However, because of the elimination of the correction factor, this method cannot correct for any other errors which come from the viscometer itself and the measuring procedure, such as, the determination of a stiffness parameter, inertial damping and inertial moment of the oscillation system, or data acquisition, curve-fit of a harmonical function, nonlinearity of oscillation of the system, or turbulent flow in the liquid. These effects also can introduce error in the viscosity measurement they cannot be corrected in the working equation. Thus clear understanding of the theory of the oscillating cup is required to obtain reliable data.

In this paper, a working equation without a correct factor is developed. The detail of the equation will be given by motion analysis of the system to define the relationship between viscosity and damping oscillation motion parameters.

2. Motion Analysis

In an oscillating cup system, a cup with a viscous liquid is suspended by an elastic wire. The cup is forced to rotate through an angle along the wire axis and then held motionless (see Fig. 1). When the cup is released, it will freely oscillate due to an elastic force which comes from the suspended wire. If the oscillation is considered as a simple harmonic motion, the two oscillation motion parameters, oscillation frequency ω and damping parameter Δ, can be measured by a curve fit technique. The viscosity of an experimental liquid can be calculated from the two motion parameters, other physical parameters of the system and sizes of the sample.

3

For an empty oscillating cup, a simple harmonic oscillation can be described as:

$$\alpha(t) = \alpha_0(t)e^{-\Delta_0\omega_0 t}\sin(\omega_0 t + \phi) \tag{1}$$

where $\alpha(t)$ is an angular displacement of the body from equilibrium and $\alpha_0(t)$ is the initial angular displacement, ϕ is an oscillatory phase shift, ω_0 is an angular frequency of the cup system without the liquid, *and* Δ_0 is a logarithmic decrement of the amplitude of the oscillation without the viscous liquid, which is caused by the internal friction of the wire and the resistance   in   the
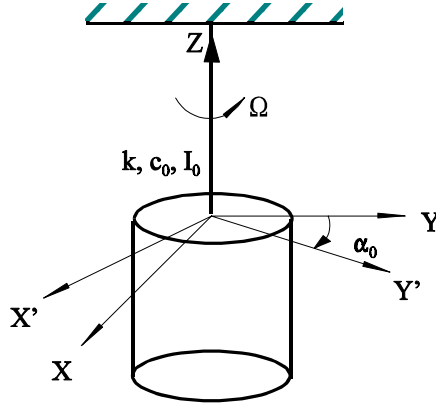
surrounding air.

Fig. 1 A torsional pendulum

Based on mechanical dynamics, the dynamic equation of the cup without liquid can be written as:

$$I_0\omega_0^2\left[\frac{d^2\alpha(\tau)}{d\tau^2} + 2\Delta_0\frac{d\alpha(\tau)}{d\tau} + (1+\Delta_0^2)\alpha(\tau)\right] = 0 \tag{2}$$

with initial conditions,

$$\text{for } t = 0, \quad \alpha(t) = \alpha_0 \text{ and } \frac{d\alpha(t)}{dt} = 0.$$

The angular frequency and damping parameter are:

4

$$\omega_0^2 = \frac{k}{I_0} - \left(\frac{c_0}{2I_0}\right)^2 \tag{3}$$

$$\Delta_0 = \frac{c_0}{2I\omega_0} \tag{4}$$

where I_o, c_o, k are respectively an initial moment of the cup, a damping coefficient of the suspended wire and a stiffness coefficient of the suspended wire. τ is a dimensionless unit of time,

$$\tau = \omega_0 t . \tag{5}$$

If the oscillating cup contains a viscous liquid, the friction force which comes from the liquid can be considered as an external applied force and put it on the right side of equation (2). So, the motion equation of the cup system with the viscous liquid becomes:

$$I\omega_0^2 \left[\frac{d^2\alpha(\tau)}{d\tau^2} + 2\Delta_0 \frac{d\alpha(\tau)}{d\tau} + (1+\Delta_0^2)\alpha(\tau)\right] = M(\tau) \tag{6}$$

By using the Laplace transform, the above motion equation is rewritten as the function of a complex frequency s.

$$[(s+\Delta_0)^2 + 1]\overline{\alpha}(s) - \frac{\overline{M}(s)}{I\omega_0^2} = (s+2\Delta_0)\alpha_0 \tag{7}$$

Where $\overline{\alpha}(s)$ and $\overline{M}(s)$ are respectively the transforms of $\alpha(\tau)$ and $M(\tau)$.

If the liquid inside the oscillating cup is considered as an ideal viscous fluid, the rate of flow is a function of the stress. The ratio of applied shearing stress to the rate of shear for an ideal viscous body is called the coefficient of viscosity, η_D .

$$\eta_D = \delta s / \delta\sigma \equiv \delta(F / A) / \delta(-dv / dz) \tag{8}$$

If the viscous body is a Newtonian fluid, the coefficient of viscosity, η , is a constant. The

5

viscosity can also be expressed by a relative kinematic viscosity, μ

$$\mu = \eta / \rho \tag{9}$$

The total friction in the liquid forms a torque, $M(\tau)$, to apply on the cup system. Its Laplace transform is:

$$\overline{M}(s) = \rho\mu\iint_A r^2 \frac{\partial\overline{\Omega}}{\partial n}d\sigma \tag{10}$$

in which, A denotes the surface of contact between the liquid and the oscillating cup, n is the normal direction of the fluid motion.

The equation of motion of the liquid is described by the Navier-Stokes equation:

$$\rho\frac{\partial\vec{u}}{\partial t} + \vec{u}\cdot\nabla\vec{u} = \rho g - \nabla p + \mu\Delta\vec{u} \tag{11}$$
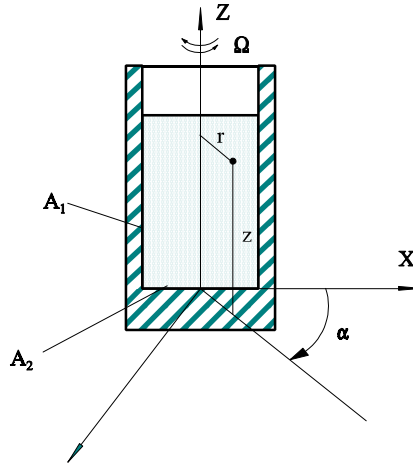

Fig. 2 Fluid flow in an oscillating cup

We usually assume that no secondary motion is developed, i.e., we omit the nonlinear terms in the Navier-Stokes equations. Based on this assumption, Equation (11) for cylindrical polar coordinates, α, r and z(see Fig. 2) is rewritten as:

6

$$\frac{\partial \Omega}{\partial t} = \mu \left\{ \frac{\partial^2 \Omega}{\partial r^2} + \frac{3}{r} \cdot \frac{\partial \Omega}{\partial r} + \frac{\partial^2 \Omega}{\partial z^2} \right\} \tag{12}$$

where Ω is an angular velocity around the z axis. The boundary conditions to be satisfied by Ω are:

-- initial condition: $t = 0$, $\quad \Omega(r, z, t) = 0$. The fluid is initially at rest.

-- boundary conditions: $r = R$, $\Omega(r, z, t) = d\alpha / dt$. There is no slip at the boundary

between
the fluid and the cup. α is an angle of the pendulum system from its equilibrium

position.

It is convenient to get dimensionless space coordinates by using an average boundary

layer thickness, $\delta = \sqrt{\mu / \omega_0}$. The motion equation (12) is rewritten as one with no unit

variables, $\xi = r / \delta$ and $\eta = z / \delta$,

$$sw = \frac{\partial^2 w}{\partial \xi^2} + \frac{3}{\xi} \cdot \frac{\partial w}{\partial \xi} + \frac{\partial^2 w}{\partial \eta^2} \tag{13}$$

where w is another non-dimensional variable,

$$w = \frac{\overline{\Omega}}{\omega_0 (s\overline{\alpha} - \alpha_0)} \tag{14}$$

Equation (14) also results in a simplified boundary condition

$$w(\xi, \eta, s) = 1 \quad on \ A$$

Equation(13) is a partial differential equation with multi-variables. It can be solved by a well-known separation of variables technique [9].

$$w(\xi, \eta, s) = \frac{\cosh \sqrt{s}(\eta_0 - \eta)}{\cosh \sqrt{s}\eta_0} + \sum_{m=0}^{\infty} \frac{\xi_0 I_1(s_m \xi)}{\xi I_1(s_m \xi_0)}(\xi, s) \sin \frac{(2m+1)\pi\eta}{2\eta_0} \tag{15}$$

7

with

$$s_m^2 = s + \left(\frac{(2m+1)\pi}{2\eta_0} \right)^2 \tag{16}$$

where I_1 denotes a Bessel function of first order.

3 Viscosity estimation equation:

After the solution of the equation of motion for liquid flow in an oscillating cup is obtained, the friction force can be calculated using Equation (9). Substituting the force into the system motion equation, we get the system equation as the following formula,

$$\frac{\overline{\alpha}(s)}{\alpha_0} = \frac{1}{s} - \frac{1 + \Delta_0^2}{s[(s+\Delta_0)^2 + 1 + D(s)]}, \tag{17}$$

with

$$D(s) = + \frac{\rho\delta^4}{I} s \iint_A \xi^2 \frac{\partial\omega}{\partial h} d\sigma \tag{18}$$

Using the solution of the motion equation (15), we can get an analytic equation for $D(s)$

$$D(s) = s^2 \frac{I'}{I} \left\{ \frac{\tanh(\sqrt{s}\eta_0)}{\sqrt{s}\eta_0} + \frac{32s}{\pi^2\xi_0} \sum_{m=0}^{\infty} \frac{1}{(2m+1)^2 s_m^3} \cdot \frac{I_2(s_m\xi_0)}{I_1(s_m\xi_0)} \right\} \tag{19}$$

By inversion of the Laplace transform, angular position now can be written as the integral

$$\alpha(\tau) = \frac{\alpha_0}{2\pi i} \int_C e^{s\tau} \left\{ \frac{1}{s} - \frac{1 + \Delta_0^2}{s[(s+\Delta_0)^2 + 1 + D(s)]} \right\} ds \tag{20}$$

along any vertical contour C in the right-hand half of the complex plane.

Equation (20), at least, can be evaluated by **residue theory** since the only singularities of integrand are poles [9]. If we get the k roots, S_k of the following equation:

$$(S_k + \Delta_0)^2 + 1 + D(S_k) = 0 \tag{21}$$

we can solve equation (20) by

8

$$\alpha(\tau) = -\alpha_0 \sum_k \frac{(1 + \Delta_0^2) \exp(S_k \tau)}{S_k [2 S_k + 2 \Delta_0 + D(S_k)]} \tag{22}$$

The ultimate purpose is to deduce the value of viscosity from the observed behavior of the oscillation. Assuming the oscillation of the cup system with viscous liquid is very nearly a damped harmonic motion, the motion, $\alpha(\tau)$, can be of a form of simple harmonic motion plus a fast decay transient motion $f(t)$.

$$\alpha(\tau) = \alpha_0 e^{-\Delta\theta\tau} \sin(\theta\tau + \phi) + f(\tau) \tag{23}$$

where $\theta = \omega / \omega_0$. After few oscillation cycles, the experimental system will behave as a simple harmonic oscillation.

$$\alpha(t) \approx e^{-\Delta\theta\tau} \cos(\theta\tau + \phi) \tag{24}$$

which responses a main pole S_k,

$$S_k = \theta(-\Delta \pm i) \tag{25}$$

Substituting above solution into equation (21), we obtain two real equations by taking the real and imaginary parts,

$$\mathrm{Re}\, D\left[\theta(-\Delta \pm i)\right] = -1 + \theta^2 - (\Delta\theta - \Delta_0)^2 \tag{26}$$

$$\mathrm{Im}\, D[\theta(-\Delta \pm i)] = \pm 2\theta(\Delta\theta - \Delta_0) \tag{27}$$

and

$$D[(-\Delta \pm i)\theta] = (-\Delta \pm i)^2 \theta^2 \frac{I}{I_0} \left\{ \frac{\tanh(\sqrt{(-\Delta \pm i)\theta\eta_0})}{\sqrt{(-\Delta \pm i)\theta\eta_0}} \right.$$

$$\left. + \frac{32(-\Delta \pm i)\theta}{\pi^2 \xi_0} \sum_{m=0}^{\infty} \frac{1}{(2m+1)^2 s_m^3} \frac{I_2(s_m \xi_0)}{I_1(s_m \xi_0)} \right\} \tag{28}$$

9

Torklep [12] used three lowest terms of the Bessel expansion and two lowest terms of *tanh* function to give a simple and approximate viscosity estimation formula,

$$D(s) = s^2 \frac{I'}{I} \left(\begin{array}{c} \dfrac{4}{s^{1/2}\xi_0} - \dfrac{6}{s\xi_0^2} + \dfrac{3}{2s^{3/2}\xi_0^3} + \dfrac{3}{2s^2\xi_0^4} + \dots \\[2mm] + \dfrac{1}{s^{1/2}\eta_0} - \dfrac{16}{\pi s\xi_0\eta_0} + \dfrac{9}{s^{3/2}\xi_0^2\eta_0} - \dfrac{8}{\pi s^2\xi_0^3\eta_0} + \dots \end{array} \right) \tag{29}$$

Substituting the equation (29) into equations (26) and (27), Torklep obtained a simplified viscosity estimation formula

$$\frac{I'}{I}[-A(\Delta p + q)\theta^{1/2}\xi_0^{-1} + B\Delta\theta\xi_0^{-2} + Cp\,\theta^{3/2}\xi_0^{-3} + D\,\theta^2\xi_0^{-4}]$$
$$= -1/\theta^2 + 1^2 - (\Delta - \Delta_0/\theta)^2 \tag{30}$$

$$\frac{I'}{I}[A(p - \Delta q)\theta^{1/2}\xi_0^{-1} - B\theta\xi_0^{-2} + Cq\,\theta^{3/2}\xi_0^{-3}] = 2\omega(\Delta\omega - \Delta_0) \tag{31}$$

where

$$A = 4 + R/H$$
$$B = 6 + (16/\pi)(R/H)$$
$$C = (3/2) + 9(R/H)$$
$$p = 1/\{2[\Delta + (1+\Delta^2)^{1/2}]\}^{1/2}$$
$$q = 1/2p$$
$$\theta = \omega/\omega_0 = T_0/T$$
$$\xi_0 = R(2\pi\rho/\eta_D T)^{1/2}$$
$$I' = \pi\rho h R^4/2$$

When the dimensionless radius, ξ_0 is larger than 10, the approximate error of Torklep's simplified equation (Equ. 30 and 31) is less than 0.1%. This requirement can be satisfied for many oscillating cup viscometers. But for some viscometers, if ξ_0 is less than 10, Torklep's equation will result some additional calculation error. In the case, it is recommended to use equation (26 - 28), which still have high calculation accuracy for any value of the

dimensionless radius.

4 Application

Figure 3 shows an oscillating cup viscometer at Auburn University. In the system, an inertia bar with a crucible is suspended with a single 56cm long and .254 mm diameter steel string. Solid samples were placed in the bottom of flat-bottomed graphite crucibles. Torsional impulses to the oscillator for initial excitation were generated through a rotary vacuum feed through by a computer-driven stepping motor at the top of the system. A HeNe laser is reflected from a mirror mounted on the inertia bar/crucible assembly, and the oscillations of the reflected laser beam are detected by two photodiodes at fixed angular positions. The working vacuum chamber is pumped with a diffusion pump from the flange shown. A temperature-controlled furnace is used to heat the alumina retort tube and provide heat energy to melt the sample. Two Pt-10% Rh thermocouples, axially spaced outside the crucible at top and bottom of the sample, are used to ensure an axial temperature uniformity on the test sample.

The system is initially at off equilibrium position 5 degrees and motionless. When a test begins, a stepping motor at the top of the pendulum quickly returns the pendulum to equilibrium position, resulting in oscillation with an initial angle of 5 degrees and with initial velocity is zero. The oscillating motion data were collected by a PC computer. A curve-fit is used to fit measured timing-position data to a simple harmonic oscillation equation (Eq. 25) and obtain the two main motion parameters, a logarithmic decrement and an oscillation period.

Previous experiments established the capability of the apparatus to characterize the viscosity of molten nickel-based superalloys. The instrument is recently improved to measure low density and low viscosity molten aluminum alloys. The following improvements have been performed on the instrument.
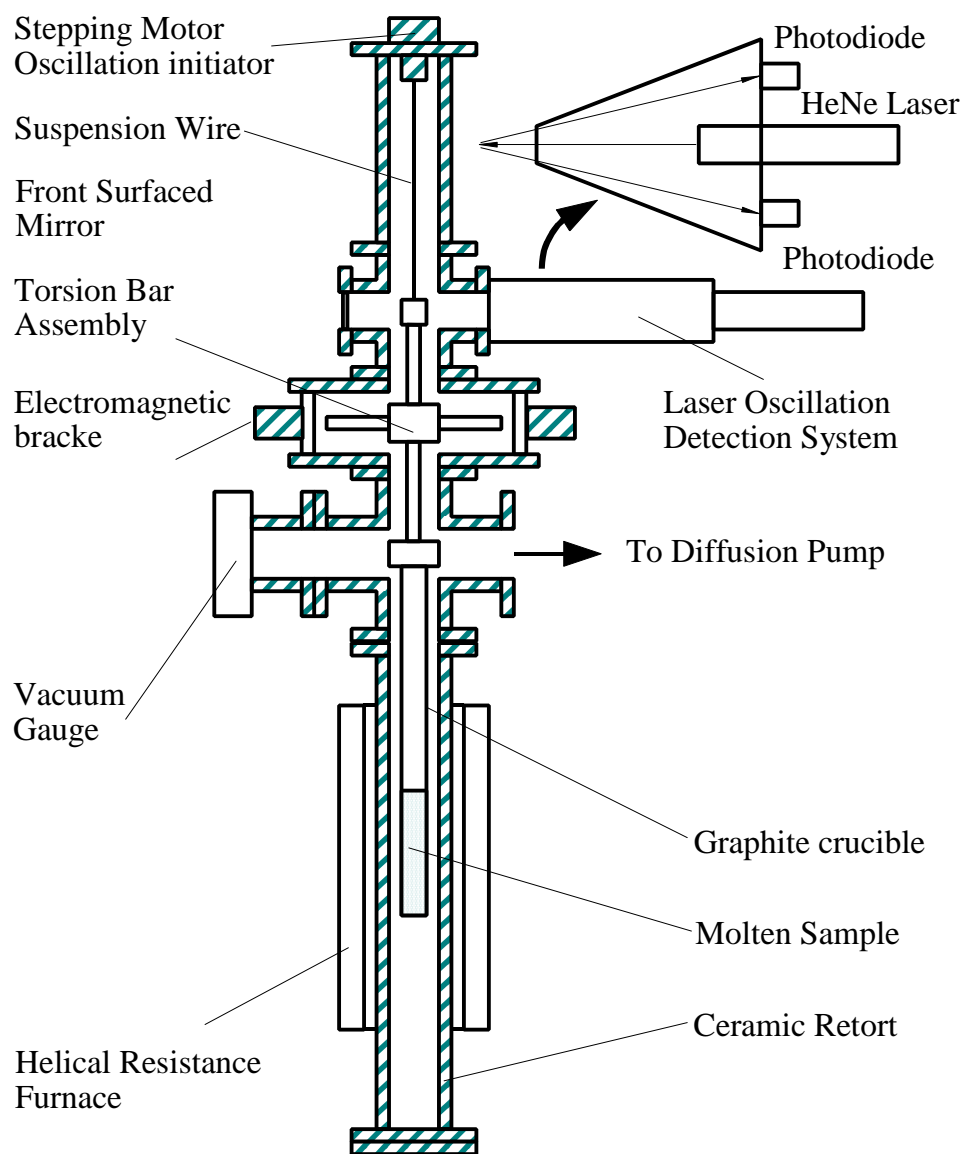
Stepping Motor
Oscillation initiator

Photodiode

HeNe Laser

Suspension Wire

Front Surfaced
Mirror

Photodiode

Torsion Bar
Assembly

Electromagnetic
bracke

Laser Oscillation
Detection System

Vacuum
Gauge

To Diffusion Pump

Graphite crucible

Molten Sample

Ceramic Retort

Helical Resistance
Furnace

Fig. 3 Schematic of the oscillating cup viscometer utilized in this study

12

– A new viscosity estimation model without a correction factor is used to calculate the viscosity from measured motion parameters, which, at least, avoids the error which comes from mathematical process (when $\xi_0 < 10$) , the calculation error is controlled at less than 0.01% for our viscometer.

– Length of a sample is increased from 5 cm into 12 cm. The larger dimension of the sample increases the rate of Δ /Δ_0 in equations (30, 31) and which ensure the calculation of equation is convergent and accurate.

– A vertical furnace was used to uniformly heat the longer cylinder sample with temperature difference controlled below 1 $^{\circ}$C. This also avoid the buoyant force which induces the second order terms in the Navier- Stokes equation and causes the simplified liquid flow calculation model(Eq. 11) to be incorrect.

– The system dynamic parameters are accurately remeasured. Before, some parameters' errors can be accomadated by a correction factor in Roscoe's working equation. Now these parameters must be accurately determinated before using the working equation (without a correction factor). Thermal effects on these parameters are considered.

– The viscosities of aluminum alloys are quite low, so the decay of oscillation is much slower. In order to record this change, more oscillation cycles are used to get the amplitude decrement of the oscillation. The measurement time increased from 100 seconds to 400 seconds, which ensures the accuracy of the curve-fit.

After improving the viscometer and adopting the working equation without a correction factor, the accuracy and repeatability are much better than before for low viscosity and low density aluminum alloy samples. Fig. 4 shows experimental data from the improved oscillating cup viscometer. Three measurements were made at each temperature, and the average viscosity data are presented.

The liquidus temperatures of these alloys were determined by a DSC instrument at Auburn University, which ensured the measured viscosities are in liquid zones of the alloys. The liquid flows of alloys in mushy zones will appear non-Newtonian. In the case, equation (9) is not satisfied, so the viscosities of two phase fluids can not be obtained by the above method. Another technique is being investigated at Auburn University to measure viscosities of metals in the mushy zone. Densities of the molten alloys were characterized in a separate investigation.

Conclusion

A working equation to calculate the viscosity of fluids from measured oscillating motion parameters without any correction factor was developed for the improved oscillating cup viscometer in Auburn University. The technique was utilized to measure viscosities of molten aluminum alloy samples with low densities and low viscosities. The experimental data are very repeatable. The study of the working equation shows it has very high mathematical accuracy, but some special attention should be taken when obtaining oscillation parameters and other system design parameters. Otherwise, the errors which come from these parameters will result in errors that cannot be calibrated in the viscosity estimation process by using the working equation.

Acknowledgment

Reference

1   W. Brockner, K. Torklep and H. A. Oye, Ber Bunsenges. Phys. Chem. **83**:1 (1979)

2   P. Banerjee and R. A. Overfelt, International Journal of Thermophysics, **20**:1791 (1999).

3   V.M.B. Nunes, F. J. V. Santos and C.A. Nieto de Castro, International Journal of Thermophysics, **19**:427 (1998).

4   R.A. Overfelt, C.A. Matlock and M.E. Wells, Metallurgical and Materials Transactions: B, **27B**:698 (1996).

5   B. Knapstad, P.A. Skjolsvik and H. A. Oye, Ber Bunsenges. Phys. Chem. **94**:1156 (1990).

6   M.R. Hopkins and T. C. Toye, Proc. Phys. Soc. B63:773 (1950)

7   Roscoe, R., Proc. Phys. Soc., **72**:576 (1958).

8   R. Roscoe and Bainbridge, Proc. Phys. Soc. **72**:585 (1958)

9   Joseph Kestin and Geodon Frank Newell, Providence, R. I., Z. Angew. Math. Phys. **8**: 433 (1957)

10   David Alan Beckwith and Gordon Frank Newell, Providence, R. I., Z. Angew. Math. Phys. **8**:450 (1957)

11   T. Ohta, O. Borgen, W. Brockner, D. Fremstad, K. Grjotheim, K. Torklep and H. A. Oye, Ber. Bunsenges Phyys. Chem. 79:335 (1975)

12   K. Torklep and H. A. Oye, J. Phys. E Sci. Instrum. **12**:875 (1979)